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Title: Protocols for Uranium Carbon Analysis - Testing Protocol Summary

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# Protocols for Uranium Carbon Analysis – Testing Protocol Summary

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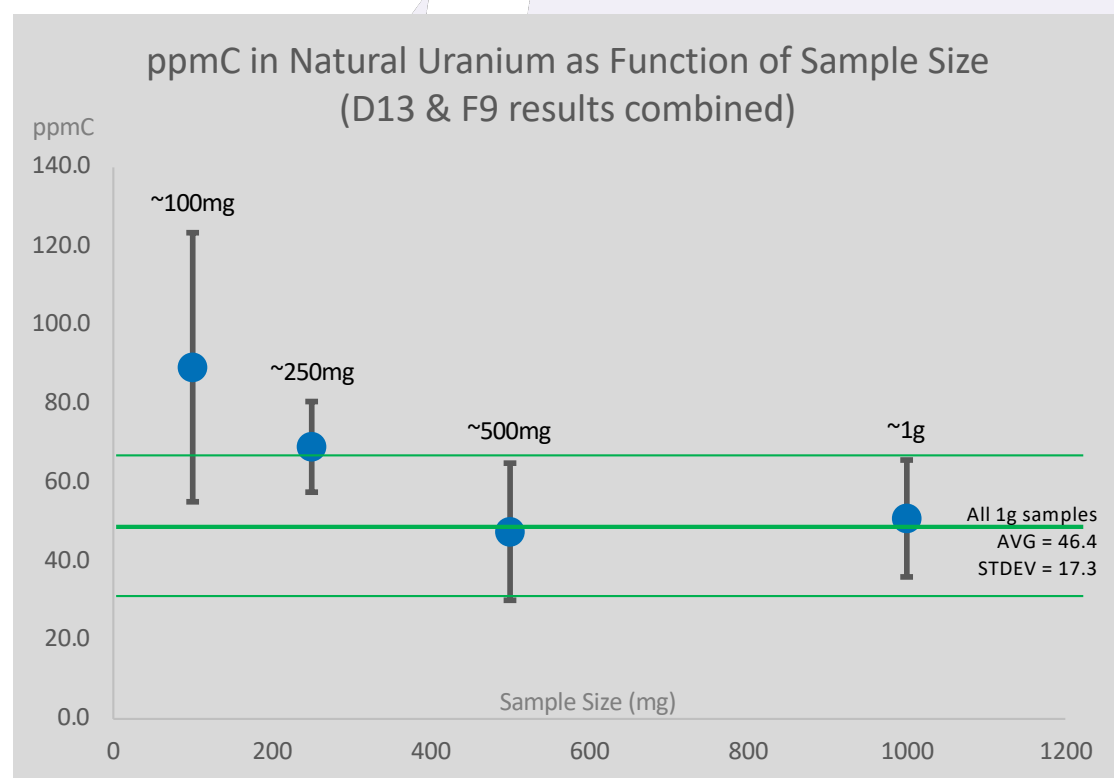
# Carbon Analysis in Uranium Protocol History

- In 1998, Sigma (then MST-6) purchased and installed a new carbon-sulfur analyzer in G105 as part of a substantial refresh of our analytic capabilities.
  - Horiba EMIA-8200W Carbon/Sulfur Analyzer
- Substantial effort was undertaken to establish analysis protocols for uranium and other materials. This summary describes the following protocols we put in place as a result of these studies.
  - Sample Size (for monolithic samples—chip and powder sample may need more current study)
  - Flux/Accelerant Recipe
  - Sample Cleaning Process

This summary is extracted from the protocol development description detailed in LA-UR-21-23989

# Uranium Sample Size Protocol for Carbon Analysis

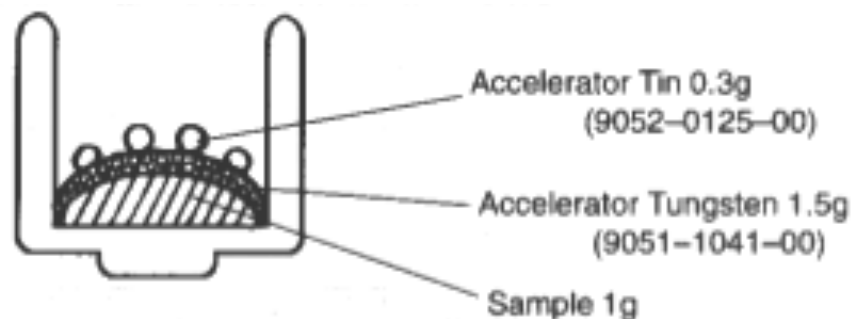
Based upon the results of >100 samples from a natural uranium carbon analysis round robin, we established **~500mg** as the lower threshold for sample size.



These results are for monolithic samples.

Some work was done on uranium chips & turnings, but not enough to establish sample size thresholds.

# Fluxing Recipe for Uranium Carbon Analysis



Flux (accelerant) recipe Sigma used  
for nearly all carbon analysis samples  
(of any sample material) <sup>1</sup>

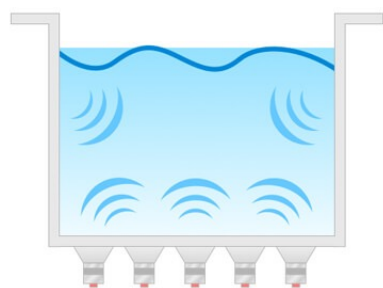
**Sample + 1.5g W + 0.3g Sn**

2.0g W + 0.5g Sn was tried. Our recollection is that this caused unacceptable crucible boil over, and splattering of the furnace tube interior.

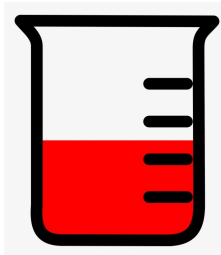
**0.5g W + 0.1g Sn "Light Flux"** was also tried for samples ~0.25g and less. This appeared to improve the accuracy of the average values, but significantly increased the spread of the data.

<sup>1</sup> Image taken from Horiba EMIA-8200W Instruction Manual, Second Edition, Horiba LTD, December 1997, Code I042935100, Page 51

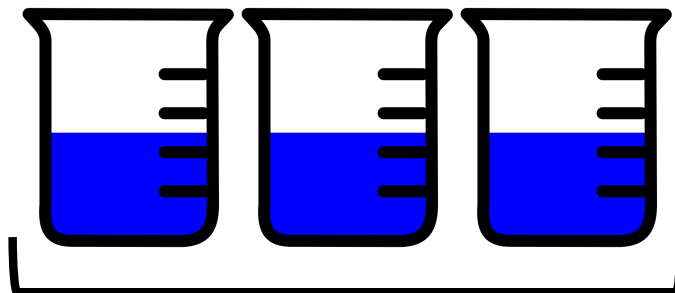
# Uranium Sample Preparation Protocol for Carbon Analysis



Soapy Water  
Ultrasonic bath  
60 sec



10% Dilute  
Nitric Acid  
90 sec <sup>1</sup>



3-Step De-Ionized  
Water Rinse Cascade  
20 sec each



Acetone  
Rinse  
5-10 sec <sup>2</sup>



Dry with  
“Warm Wind” <sup>3</sup>

<sup>1</sup> A 5:1 Concentrated Nitric Acid cleaning for 15-20 seconds was also used on occasion

<sup>2</sup> If fully dried/evaporated from the sample surface, acetone was shown not to affect the carbon results

<sup>3</sup> This term was taken from one of the Horiba EMIA manuals. We utilized a standard commercial electric heat gun to dry the samples.